

PRINTER RUSH

(PTO ASSISTANCE)

Application : W/576694 Examiner : Gale GAU : 1621
From: MR Location: IDC FMF FDC Date: 05-01-88
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Date:

DOC CODE	DOC DATE	MISCELLANEOUS
<input type="checkbox"/> 1449		<input type="checkbox"/> Continuing Data
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<input checked="" type="checkbox"/> SPEC	<u>04-21-86</u>	

[RUSH] MESSAGE: Page 17 of the specification is
missing, please supply
Thank you
MR

[XRUSH] RESPONSE: Page 17 is attached
DONE! PLEASE SEE ATTACHED. KG
INITIALS: KG

EXAMINER: PUBS contacts -- for DESIGNS: Don Fairchild, 703-308-9250 x126; for ALL OTHER FILES:
Bernadette Queen, 703-308-9250 x121

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PF 55020

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heated to a temperature according to table 1. 6.1 g (50 mmol) of 2,4-dimethylphenol were then metered in. The suspension consisted of flocks and was readily stirrable.

- 5 A certain volume (cf. table 1) of a 19% by weight aqueous sodium persulfate solution (1 mole equivalent/180 min) was then metered in by means of a pump (Metrohm-Dosimat 665) in the course of the time stated in table 1. After the end of the addition, the mixture was stirred for a further 4 hours. The suspension was cooled to room temperature and 199 ml of toluene was added. The three-phase suspension was filtered over a suction filter. The organic phase was washed with water and evaporated
10 down. The residue was analyzed by means of gas chromatography, and conversion, selectivity and yield were determined by means of a standard (cf. table 1).

Comparative example (according to US 6077979)

- 15 4.18 g (15 mmol) of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in 450 ml of water were initially taken in a 1 000 ml flask which carried a thermometer and was equipped with a Teflon paddle stirrer (350 revolutions per minute). 36.8 g (300 mmol) of 2,4-dimethylphenol were then metered in.
- 20 An aqueous sodium persulfate solution (71.5 g of $\text{Na}_2\text{S}_2\text{O}_8$ in 300 ml of water) was metered into the two-phase mixture by means of a pump (Metrohm-Dosimat 665) in the course of 300 minutes at room temperature.

- 25 After some time, a greasy solid formed, some of which adhered to the reactor wall and the stirrer and did not permit effective mixing of the reaction mixture. After the end of the addition, the mixture was stirred for a further 3 days. In the course of this time, the greasy precipitate became hard.

Table 1

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Exp.	Feed time [min]	Peroxide/phenol. [mol/mol]	Temperature [°C]	Conversion [%]	Selectivity [%]	Yield [%]
1	180	1.0	60	100	53	53
2	180	1.0	40	94	75	71
3	90	0.5	60	91	74	67
4	90	0.5	50	90	77	69
5	90	0.5	40	89	77	69